

Environmental Technology Verification Coatings and Coating Equipment Program (ETV CCEP)

Final Technology Applications Group TAGNITE – Testing and Quality Assurance Plan (T/QAP)

July 24, 2006

Distribution Statement “A” applies
Approved for public release; distribution is unlimited

Requests for this document shall be referred to:

Office of the Assistant Secretary of the Army
for Installations and Environment
ASA (I&E) - ESOH
1235 Clark Street
Crystal Gateway 1, Suite 307
Arlington, VA 22202-3263

Contract No. W74V8H-04-D-0005
Task No. 0428
CDRL No. A004

*Prepared by
National Defense Center for Environmental Excellence (NDCEE)*

*Submitted by
Concurrent Technologies Corporation
100 CTC Drive
Johnstown, PA 15904*

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Washington Headquarters Services, Directorate for information operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE 24 July 06		3. REPORT TYPE AND DATES COVERED Final (Sep. 2005 – Nov. 2006)
4. TITLE AND SUBTITLE Technology Applications Group TAGNITE – Testing and Quality Assurance Plan (T/QAP); Revision No. 0			5. FUNDING NUMBERS Contract: W74V8H-04-D-0005 Task: NDCEE Task No. 0428	
6. AUTHOR(S) Principal Author/PM: Robert J. Fisher, CTC				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Defense Center for Environmental Excellence Operated by Concurrent Technologies Corporation 100 CTC Drive Johnstown, PA 15904			8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) NDCEE Program Office (Office of the Assistant Secretary of the Army for Installations and Environment) 1235 Clark Street, Suite 307 Arlington, VA 22202-3263			10. SPONSORING/MONITORING AGENCY REPORT NUMBER NDCEE-CR-2006-058	
10 SUPPLEMENTARY NOTES				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) <p>The ETV CCEP verifies the pollution prevention and performance characteristics of coating technologies and make sthe results of the testing available to prospective coating technology users. The objective of this particular Testing and Quality Assurance Plan (T/QAP) is to establish the performance of the Technology Applications Group (TAG) TAGNITE magnesium anodizing process. This innovative process was developed and patented by TAG to replace other anodizing processes and conversion coatings for magnesium alloys. The test data from this verification test will be compiled and used to develop a Verification Report, and, at the discretion of the vendor, a Verification Statement will be developed from the data contained in the Verification Report. TAG may use the Verification Statement as a marketing tool for the TAGNITE process, in accordance with the ETV Program requirements.</p> <p>The primary purpose of this document is to establish the T/QAP for the TAGNITE magnesium anodizing process, and to establish specific data quality requirements for the verification of this technology. The vendor claims that the TAGNITE process is capable of exceeding the performance characteristics of existing magnesium anodizing processes while providing and environmental benefit in terms of eliminating the generation of chromates or</p>				
14. SUBJECT TERMS			15. NUMBER OF PAGES 63	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT None	

NSN 7540-01-280-5500

Standard Form 298 Rev. 12/00
Prescribed by ANSI ST. 239-18
880922

TABLE OF CONTENTS

	Page
SI TO ENGLISH CONVERSIONS	iv
LIST OF ABBREVIATIONS AND ACRONYMS	v
1.0 INTRODUCTION.....	1
1.1 Purpose of the Technology Applications Group TAGNITE® - T/QAP	1
1.2 Quality Assurance for the ETV CCEP	1
1.3 Organization of the TAGNITE T/QAP	1
1.4 Formatting	2
1.5 Approval Form.....	2
2.0 PROJECT DESCRIPTION.....	3
2.1 General Overview	3
2.1.1 Off-Site Panel Preparation Phase.....	3
2.1.2 CTC's Environmental Coatings Laboratory.....	3
2.1.3 Statement of Project Objectives	5
2.2 Technical/Experimental Approach and Guidelines.....	5
2.2.1 Test Approach.....	6
2.2.2. Verification Test Objectives.....	7
2.2.3 Standard Test Panels.....	7
2.2.4 Galvanic Corrosion Coupons.....	7
2.2.5 Process Standards	7
2.2.6 Design of Experiment	9
2.2.7 Performance Testing.....	10
2.2.8 Quantitative Measurements.....	10
2.2.9 Participation.....	10
2.2.10 Critical and Non-Critical Factors.....	11
2.3 Schedule.....	13
3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES	15
4.0 QUALITY ASSURANCE OBJECTIVES.....	18
4.1 General Objectives.....	18
4.2 Quantitative Quality Assurance Objectives.....	18
4.2.1 Accuracy.....	20
4.2.2 Precision.....	21
4.2.3 Completeness	21
4.2.4 Impact and Statistical Significance Quality Objectives.....	21
4.3 Qualitative QA Objectives: Comparability and Representativeness	22
4.3.1 Comparability	22
4.3.2 Representativeness.....	22

4.4	Other QA Objectives.....	22
4.5	Impact of Quality	22
5.0	SITE SELECTION AND SAMPLING PROCEDURES.....	24
5.1	Site Selection.....	24
5.2	Sampling Procedures and Handling.....	24
5.3	Sample Custody, Storage and Identification.....	24
6.0	ANALYTICAL PROCEDURES AND CALIBRATION.....	26
6.1	Facility and Laboratory Testing and Calibration.....	26
6.1.1	Facility Testing and Calibration.....	26
6.1.2	Laboratory Testing and Calibration Procedures.....	26
6.2	Product Quality Procedures.....	27
6.3	Standard Operating Procedures and Calibration.....	27
6.4	Non-Standard Methods	28
7.0	DATA REDUCTION, VALIDATION, AND REPORTING	29
7.1	Raw Data Handling	29
7.2	Preliminary Data Package Validation	29
7.3	Final Data Validation.....	29
7.4	Data Reporting and Archival.....	29
7.4.1	Calculation of DFT Variation.....	29
7.4.2	Evaluation of the Technology Applications Group, Inc. TAGNITE Magnesium Anodizing System.....	30
7.5	Verification Statement	30
8.0	INTERNAL QUALITY CONTROL CHECKS.....	31
8.1	Guide used for Internal Quality Program	31
8.2	Types of QA Checks	31
8.3	Basic QA Checks	31
8.4	Specific Checks	33
9.0	PERFORMANCE AND SYSTEM AUDITS	34
10.0	CALCULATION OF DATA QUALITY INDICATORS	35
10.1	Precision.....	35
10.2	Accuracy.....	35
10.3	Completeness	36
10.4	Project Specific Indicators.....	36
11.0	CORRECTIVE ACTION	37
11.1	Routine Corrective Action.....	37
11.2	Non-Routine Corrective Action.....	37
12.0	QUALITY CONTROL REPORTS TO MANAGEMENT.....	38

13.0	REFERENCES	39
------	------------------	----

LIST OF TABLES

Table 1. Overall Guidelines and Procedures to be Applied to this T/QAP	6
Table 2. Test Matrix Summary	9
Table 3. Critical Control Factors	12
Table 4. Non-Critical Control Factors	12
Table 5. Critical Response Factors	13
Table 6. Estimated Schedule as of 05/05/06	14
Table 7. Summary of ETV CCEP Experience and Responsibilities	16
Table 8. Frequency and Mechanisms of Communications	17
Table 9. Responsibilities During Testing	17
Table 10. QA Objectives for Precision, Accuracy and Completeness for All Non-Critical Control Factor Performance Analyses.....	19
Table 11. QA Objectives for Precision, Accuracy and Completeness for All Critical Response Factor Performance Analyses.....	20
Table 12. Process Responsibilities	24
Table 13. Non-Critical Control Factor Testing and Calibration Criteria.....	27
Table 14. Critical Response Factor Testing and Calibration Criteria.....	28
Table 15. CTC Laboratory QA/QC Format Sources.....	31

LIST OF FIGURES

Figure 1. Project Organization Chart.....	15
---	----

LIST OF APPENDICES

A	Standard Test Panels
B	Galvanic Corrosion Coupons
C	TAGNITE Product Specifications
D	Baseline Treatments' Product Specifications
E	ASTM Methods

SI to English Conversions

SI Unit	English Unit	Multiply SI by factor to obtain English
°C	°F	1.80, then add 32
L	gal. (U.S.)	0.2642
m	ft	3.281
kg	lbm	2.205
kPa	psi	0.14504
cm	in.	0.3937
mm	mil (1 mil = 1/1000 in.)	39.37
m/s	ft/min	196.9
kg/L	lbm/gal. (U.S.)	8.345

List of Abbreviations and Acronyms

ANSI	American National Standards Institute
AOAC	Association of Official Analytical Chemists
ASME	American Society of Mechanical Engineers
ASQC	American Society for Quality Control
ASTM	American Society for Testing and Materials
CCEP	Coatings and Coating Equipment Program
CTC	Concurrent Technologies Corporation
DFT	dry film thickness
EPA	U.S. Environmental Protection Agency
ETF	Environmental Technology Facility
ETV	Environmental Technology Verification
HAP	hazardous air pollutant
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectrometry
ID	identification
ISO	International Standardization Organization
NDCEE	National Defense Center for Environmental Excellence
NIST	National Institute for Standards and Technology
OFL	Organic Finishing Lab
QA/QC	quality assurance/quality control
QMP	Quality Management Plan
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SRM	Standard Reference Materials
TAG	Technology Applications Group
T/QAP	Testing and Quality Assurance Plan
VOC	volatile organic compound

1.0 INTRODUCTION

1.1 Purpose of the Technology Applications Group TAGNITE® - T/QAP

The primary purpose of this document is to establish the Testing and Quality Assurance Plan (T/QAP) for the Technology Applications Group (TAG) TAGNITE magnesium anodizing process. The objective of this T/QAP is to verify the performance of the TAGNITE process, which is chromate and permanganate free. The format and guidelines for this T/QAP are established below.

This T/QAP establishes specific data quality requirements for all technical parties involved in the verification of the TAGNITE process. This T/QAP follows the format described below to facilitate independent reviews of the project plans and test results, and to provide a standard platform of understanding for stakeholders and participants.

1.2 Quality Assurance for the ETV CCEP

Projects conducted under the auspices of the U.S. Environmental Protection Agency's (EPA) Environmental Technology Verification Coatings and Coating Equipment Program (ETV CCEP) meet or exceed the requirements of the American National Standards Institute/American Society for Quality Control (ANSI/ASQC), Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs, ANSI/ASQC E-4 (1994) standard (see Section 13 for reference). This T/QAP will ensure that project results are compatible with and complementary to similar projects. All ETV CCEP T/QAPs are adapted from this standard and the ETV Program Quality Management Plan (QMP) (see Section 13 for reference). T/QAPs will contain sufficient detail to ensure that measurements are appropriate for achieving project objectives, that data quality is known, and that the data are legally defensible and reproducible.

1.3 Organization of the TAGNITE T/QAP

This T/QAP contains the sections outlined in the ANSI/ASQC E-4 standard. As such, this T/QAP identifies processes to be used, test and quality objectives, measurements to be made, data quality requirements and indicators, and procedures for the recording, reviewing and reporting of data.

The major technical sections discussed in this T/QAP are as follows:

- ? Project Description
- ? Project Organization and Responsibilities
- ? Quality Assurance (QA) Objectives
- ? Site Selection and Sampling Procedures
- ? Analytical Procedures and Calibration
- ? Data Reduction, Validation and Reporting
- ? Internal Quality Control (QC) Checks
- ? Performance and System Audits
- ? Calculation of Data Quality Indicators
- ? Corrective Action
- ? Quality Control Reports to Management
- ? Appendices

1.4 Formatting

In addition to the technical content, this T/QAP also contains standard formatting elements required by the ANSI/ASQC E-4 standard and Concurrent Technologies Corporation (CTC) deliverables.

1.5 Approval Form

Key ETV CCEP personnel indicate their agreement and common understanding of the project objectives and requirements by signing a T/QAP Approval Form for the verification testing of the TAGNITE process. Acknowledgment by each key person indicates commitment toward implementation of the plan.

2.0 PROJECT DESCRIPTION

2.1 General Overview

The overall objective of the ETV CCEP is to verify pollution prevention and performance characteristics of coating technologies and make the results of the testing available to prospective coating technology users. The objective of this particular T/QAP is to establish the performance of the TAGNITE magnesium anodizing process. This innovative process was developed and patented by TAG to replace other anodizing processes and conversion coatings for magnesium alloys. The test data from this verification test will be compiled and used to develop a Verification Report, and, at the discretion of the vendor, a Verification Statement will be developed from the data contained in the Verification Report. TAG may use the Verification Statement as a marketing tool for the TAGNITE process, in accordance with the ETV Program requirements.

2.1.1 Off-Site Panel Preparation Phase

TAG's facility in Grand Forks, ND, will be the location for the TAGNITE coating application portion of this verification test. The baseline anodizing, conversion coatings, sealers, primers, and topcoats will be applied per the appropriate specifications either in-house at CTC's Johnstown, PA, Environmental Technology Facility (ETF), or at an off-site location(s) familiar with the particular treatments. Whenever an off-site location is utilized for any portion of this test, the ETV CCEP staff will conduct site visits, perform technical audits, and oversee all sample preparations. The ETV CCEP staff will also measure process variables, conduct some on-site laboratory analyses, and package the Standard Test Panels and Galvanic Corrosion Coupons for transport to the Environmental Coatings Laboratory in CTC's ETF. In the event other laboratory analyses are required where a particular capability is not present at the CTC's facilities, an outside laboratory may be obtained to complete those analyses.

2.1.2 CTC's Environmental Coatings Laboratory

In support of the ETV CCEP, the extensive state-of-the-art Environmental Coatings Laboratory within CTC's ETF facility will be available to evaluate the treated and/or coated Standard Test Panels and Galvanic Corrosion Coupons. Laboratory facilities available for this test include a taber abrasion unit, multiple salt spray chambers, and a direct impact unit. The Environmental Coatings Laboratory will also conduct sample analyses of the various raw treatment chemicals and their respective waste streams to determine the level of

chromates and other heavy metals present, as well as pH and volumes used and/or generated.

2.1.3 Statement of Project Objectives

The overall objective of the ETV CCEP is to verify pollution prevention characteristics and/or performance of coatings and coating equipment technologies, and to make the results of the verification tests available to prospective technology users. The ETV CCEP aspires to increase the use of more environmentally friendly technologies in products finishing in an effort to reduce emissions.

The primary criteria for verification of TAGNITE process will be:

- ? Does the coating provide an environmental benefit in terms of reduced chromate and heavy metal generation compared to existing processes and coatings?
- ? Does the coating perform to an acceptable level of quality and performance?
- ? Is there a reduction of solid, liquid, or hazardous waste?

Based on the best available data, as presented by an unbiased third party, end-users will be able to determine whether the coating can provide them with a pollution prevention benefit while meeting the finish quality requirements of their application. This program intends to supply end-users with the unbiased technical data to assist them in the decision-making process.

The quantitative pollution prevention benefit in terms of reducing or eliminating chromates and permanganates depends on a multitude of factors; therefore, the TAGNITE process will be applied per TAG's instructions, and the resulting verification data will be representative of the exact conditions tested. To qualify the existence of an environmental benefit, this program will conduct a test to qualify the chemicals used in the process and determine if the TAGNITE process improves the durability of magnesium alloys compared to existing technologies.

2.2 Technical/Experimental Approach and Guidelines

The following tasks are planned for this project (see estimated schedule in Section 2.3, Table 6):

- ? Obtain TAG's concurrence with this T/QAP.
- ? Obtain CTC and EPA approval of this T/QAP.
- ? Conduct verification test of the TAGNITE process.
- ? Prepare and provide the draft Verification Report to EPA.
- ? Prepare and provide the final Verification Report to EPA.
- ? Prepare Verification Statement for approval and distribution.

Table 1 describes the general guidelines and procedures that will be applied to this T/QAP.

Table 1. Overall Guidelines and Procedures to be Applied to this T/QAP

?	A detailed description of each part of the test will be given.
?	Critical and non-critical factors will be listed. Non-critical factors will remain constant throughout the testing. Critical factors will be listed as control (process) factors or response (product quality) factors (see Section 2.2.10 below).
?	This T/QAP will identify the potential testing sites.
?	All testing will be under the control and close supervision of ETV CCEP representatives to ensure the integrity of the third party testing.
?	The QA portion of this T/QAP will be strictly adhered to.
?	A statistically significant number of samples will be analyzed for each critical response factor (see Table 5). Variances (or standard deviations) of each critical response factor will be reported for all results.

2.2.1 Test Approach

The following approach will be used in the test protocol.

- ? TAG and the ETV CCEP will agree on the performance parameters to be verified;
- ? TAG will supply the Standard Test Panels, Galvanic Corrosion Coupons, and related materials;
- ? TAG will provide the TAGNITE process capability;
- ? The baseline treatments, sealers, primers, and topcoats will be applied by CTC or other qualified, independent facilities;
- ? Data such as dry film thickness, abrasion resistance, salt spray, and impact resistance will be collected, following American Society for Testing and Materials (ASTM) methods, or equivalent; and,
- ? A statistically valid test program that efficiently accomplishes the required objectives will then be used to analyze the data.

2.2.2. Verification Test Objectives

The objectives of the verification test performed per this T/QAP are to determine the environmental and performance benefits that the TAGNITE process provides over a baseline of anodizing processes and conversion coatings. The coated Standard Test Panels will be tested for dry film thickness (DFT), direct impact, abrasion resistance, and salt-spray resistance (including paint adhesion, short-term corrosion pit depth analysis, and galvanic corrosion).

2.2.3 Standard Test Panels

The Standard Test Panels to be used for this verification test will consist of four magnesium alloys (ZE41A, EV31, AZ91E, and ZK60). A dimensioned drawing of the Standard Test Panels is shown in Appendix A (Standard Test Panels). TAG will obtain the required materials and provide a sufficient number of panels to CTC. The Standard Test Panels will have the same approximate dimensions of 10.2 cm (4 in.) wide, 15.2 cm (6 in.) long, and 0.6 cm (0.25 in.) thick. The Standard Test Panels will include a threaded hole on the top, short side, and an aluminum bolt, which will aid in handling the panels and as a conductive connecting point. CTC will evaluate each panel prior to treatment to ensure that the surface profiles of the panels are similar.

2.2.4 Galvanic Corrosion Coupons

The Galvanic Corrosion Coupons will consist of two magnesium alloys (ZE41A and EV31). A dimensioned drawing of the Galvanic Corrosion Coupons is shown in Appendix B (Galvanic Corrosion Coupons). TAG will obtain the required materials and provide a sufficient number of coupons to CTC. The Galvanic Corrosion Coupons will have the same approximate dimensions of 7.6 cm (3 in.) wide, 7.6 cm (3 in.) long, and 3.1 cm (1.25 in.) thick. The Galvanic Corrosion Coupons will include a threaded hole in the center of the square sides. Cadmium-plated bolts, washers, and nuts will be used to initiate the galvanic action in the salt spray chamber. As with the Standard Test Panels, CTC will check the surface profile of each Galvanic Corrosion Coupon prior to the test.

2.2.5 Process Standards

The Standard Test Panels and Galvanic Corrosion Coupons will be made of multiple alloys, as listed in Table 2. The treatments will be evaluated alone, with a sealer coating, and with a waterborne primer/topcoat system applied over a sealer coating. The anodizing process will be the similar for each alloy type and

test combinations. Operating parameters will be held relatively constant throughout the test.

2.2.6 Design of Experiment

This T/QAP will be used to verify the performance of the TAGNITE process. A mean value and variance (or standard deviation) will be reported for each critical response factor, and default 95% confidence limit will be applied. The statistical analyses for all response factors will be carried out using the latest version of Minitab statistical software.

The verification test will be comprised of sixty-four (64) separate combinations with three (3) Standard Test Panels or Galvanic Corrosion Coupons per combination. This will enable total variation to be determined for each response factor.

Table 2 shows the test matrix summary. The test samples are grouped according to their finished state (e.g., treated panels only, treated panels with sealer, treated panels with sealer, primer, and topcoat, and treated coupons with sealer). Four magnesium treatments and four magnesium alloys will be used. The number in parenthesis after each alloy represents the number of samples to be created using that particular combination. The product specifications for the four magnesium treatments can be found in Appendix C (TAGNITE Product Specifications) and D (Baseline Treatments' Product Specifications).

Table 2. Test Matrix Summary

	TAGNITE	Dow 17	H.A.E.	Dow 7
Standard Test Panels with treatment only	ZE41A (12) EV31 (9) AZ91E (3) ZK60 (3)	ZE41A (12) EV31 (9) AZ91E (3) ZK60 (3)	ZE41A (12) EV31 (9) AZ91E (3) ZK60 (3)	ZE41A (6) EV31 (6) AZ91E (3) ZK60 (3)
Standard Test Panels with treatment and sealer	ZE41A (6) EV31 (3) AZ91E (3)	ZE41A (6) EV31 (3) AZ91E (3)	ZE41A (6) EV31 (3) AZ91E (3)	ZE41A (3) EV31 (3) AZ91E (3)
Standard Test Panels with treatment, sealer, primer, & topcoat	ZE41A (3) EV31 (3)	ZE41A (3) EV31 (3)	ZE41A (3) EV31 (3)	ZE41A (3) EV31 (3)
Galvanic Corrosion Coupons with sealer	ZE41A (3) EV31 (3)	ZE41A (3) EV31 (3)	ZE41A (3) EV31 (3)	ZE41A (3) EV31 (3)

2.2.7 Performance Testing

Standard Test Panels and Galvanic Corrosion Coupons will be used to measure the performance characteristics of each magnesium treatment. The manufacture and final surface preparation will be the same for all Standard Test Panels and Galvanic Corrosion Coupons, prior to testing. The panels and coupons will be evaluated prior to testing to ensure that the variability between samples is minimal. Non-critical control factors will be monitored or held relatively constant for the verification test. A comparison will be made from combination to combination.

2.2.8 Quantitative Measurements

In order to establish a basis for comparison between the magnesium treatments, DFT will be measured on the Standard Test Panels and Galvanic Corrosion Coupons after each treatment is applied, after each layer of sealer, and after each layer of primer and topcoat. The uniformity of the applied layers will be determined by measuring DFT at several locations on each sample. There are fourteen (14) measurement sites on the Standard Test Panels, and twelve (12) sites on each Galvanic Corrosion Coupon. The sites will be numbered so that the recorded measurements can be correlated to a specific site (see Appendices A and B). The thickness measurement data will be used to evaluate the average thickness and thickness variation across the samples.

Chemical analyses will be completed of both the process solutions and the waste streams to determine the relative environmental impacts of each treatment. The chemical analyses will focus on heavy metals and other hazardous compounds, in addition to the pH during the various stages of each process. A cursory review of the materials used to make up each process solution will be conducted to determine whether there is a potential for volatile organic compound (VOC) and/or hazardous air pollutant (HAP) emissions.

2.2.9 Participation

TAG will supply the Standard Test Panels and Galvanic Corrosion Coupons. TAG will also treat the appropriate samples with TAGNITE. The ETV CCEP personnel will be responsible for verifying that all data and QA requirements have been met. The ETV CCEP personnel will also perform all laboratory analyses identified for this verification whenever possible. Off-site facilities or laboratories may be utilized when a particular capability is not available at CTC. However, ETV CCEP personnel will oversee and observe all sample preparation activities.

2.2.10 Critical and Non-Critical Factors

In a designed experiment, critical and non-critical control factors must be identified. In this context, the term "critical" does not convey the importance of a particular factor. (Importance can only be determined through experimentation and characterization of the total process.) Rather, this term displays its relationship within the design of experiments. For the purposes of this T/QAP, the following definitions will be used for critical control factors, non-critical control factors, and critical response factors.

- ? Critical control factor - a factor that is varied in a controlled manner within a design of experiments matrix to determine its effect on a particular outcome of a system.
- ? Non-critical control factors - factors that remain relatively constant or are randomized throughout the testing.
- ? Critical response factors - the measured outcomes of each combination of critical and non-critical control factors used in the design of experiments.

In this verification test, there are four critical control factors, the four separate treatment processes. All other processing factors are non-critical control factors; therefore, the multiple combinations and sample measurements within each combination of critical control factors will be used to determine the amount of variation expected for each critical response factor.

Tables 3 through 5 identify the factors to be monitored during testing, as well as their acceptance criteria (where appropriate), data quality indicators, measurement locations, and measurement frequencies. The values in the Total Numbers column are based on the default test scenarios.

Table 3. Critical Control Factors

Critical Control Factor	PH	Operating Temperature	Bath Solution	Coating Thickness
TAGNITE Magnesium Anodizing Process	12.8 – 13.2	4.4 – 15.5 °C (40 – 60 °F)	Hydroxide Silicate Fluoride	Type I: 0.2 – 0.4 mil
Dow 17 Magnesium Anodizing Process	~5	≥71 °C (≥160 °F)	Phosphate Fluoride Chromate	Type I: 0.1 – 0.5 mil
HAE Magnesium Anodizing Process	~14	21 – 30 °C (70 – 86 °F)	Hydroxide Fluoride Phosphate Manganate	Type I: 0.1 – 0.3 mil
Dow 7 Conversion Coating	4.1 – 5.6	Boiling ≥ 93 °C (≥200 °F)	Chromate Fluoride	30 to 45 min. ^a

^a The Dow 7 conversion coating is applied in a very thin film thickness. The amount of material deposited is based on the duration of the treatment process.

Table 4. Non-Critical Control Factors

Non-Critical Factor	Set Points/ Acceptance Criteria	Measurement Location	Frequency	Total Number for the Test
Products Involved in Testing	Standard Test Panels and Galvanic Corrosion Coupons	Each Standard Test Panel or Galvanic Corrosion Coupon	Once per Standard Test Panel or Galvanic Corrosion Coupon	192
Surface Area of Standard Test Panels and Galvanic Corrosion Coupons	Varies <16.1 cm ² (<2.5 in. ²)	CTC	All Standard Test Panels or Galvanic Corrosion Coupons	192
Surface Profile of Standard Test Panels and Galvanic Corrosion Coupons	ASME B46.1	CTC	All Standard Test Panels or Galvanic Corrosion Coupons	192

Table 5. Critical Response Factors

Critical Response Factor	Measurement Location	Frequency	Total Number
Environmental			
Chromate and Heavy Metal Content and pH in Total Process	Process Area	Once for each process solution and waste stream	Varies
Quality/Performance (Mandatory)			
Dry Film Thickness (DFT)	ASTM B 244 (Eddy Current)	14 points on each Standard Test Panel (12 points on each Galvanic Corrosion Coupon) per coating layer	4566
Taber Abrasion, Treatment Only	ASTM D 4060; CS-10	3 samples per combination, 6 combinations (ZE41A and EV31 with TAGNITE, Dow 17, & H.A.E.)	18
Salt Spray, Treatment Only, Scribed (Failure or Score=7)	ASTM B 117	3 samples per combination, 16 combinations (All four alloys and all four treatments)	48
Salt Spray Pit Depth, Treatment Only, Scribed (168 hours)	ASTM B 117	3 samples per combination, 8 combinations (ZE41A and EV31 with all four treatments)	24
Salt Spray, Treatment w/ Sealer, Scribed (1000 hrs or Score=7)	ASTM B 117	3 samples per combination, 12 combinations (ZE41A, EV31, and AZ91E with all four treatments)	36
Salt Spray, Treatment w/ Sealer, Primer, and Topcoat, Un-Scribed (1000 hrs or Score=7)	ASTM B 117	3 samples per combination, 8 combinations (ZE41A and EV31 with all four treatments)	24
Direct Impact, Treatment Only	ASTM D 2794	3 samples per combination, 3 combinations (ZE41A with TAGNITE, Dow 17, and H.A.E.)	9
Direct Impact, Treatment w/ Sealer	ASTM D 2794	3 samples per combination, 3 combinations (ZE41A with TAGNITE, Dow17, and H.A.E.)	9
Galvanic Corrosion Salt Spray w/ Sealer, Un-Scribed (1000 hrs or Score=7)	ASTM B 117	3 samples per combination, 8 combinations (ZE41A and EV31 with all four treatments)	24

Score = 7 refers to the amount of corrosion considered to be a failure, as determined using ASTM B 117.

2.3 Schedule

ETV CCEP uses standard tools for project scheduling. Project schedules are prepared in Microsoft Project, which is an accepted industry standard for scheduling. Project schedules show the complete work breakdown structure of the project, including technical work, meetings and deliverables. Table 6 shows the estimated schedule for the verification testing of the TAGNITE process.

Table 6. Estimated Schedule as of 07/24/06

ID	Name	Duration	Start Date	Finish Date
Task 1	Approval of T/QAP	15d	TBD	TBD
Task 2	Verification Testing	80d	TBD	TBD
Task 3	Complete Data Analyses	10d	TBD	TBD
Task 4	Prepare Verification Report	30d	TBD	TBD
Task 5	Approval of Verification Report	90d	TBD	TBD
Task 6	Issue Verification Statement	15d	TBD	TBD

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

CTC employs a matrix organization, with program and line management, to perform projects. The laboratory supports the ETV CCEP Project Manager by providing test data. Laboratory Analysts report to the ETV CCEP Laboratory Leader. The ETV CCEP Laboratory Leader and Organic Finishing Engineer coordinate with the ETV CCEP Project Manager on testing schedules. The ETV CCEP Project Manager will be responsible for preparing the T/QAPs and Verification Report and Statement for each test.

The ETV CCEP QA Officer, who is independent of both the laboratory and the program, is responsible for administering *CTC* policies developed by the Quality Committee. These policies provide for, and ensure that quality objectives are met for each project. The policies are applicable to laboratory testing, factory demonstration processing, engineering decisions, and deliverables. The ETV CCEP QA Officer reports directly to *CTC* senior management and is organizationally independent of the project or program management activities.

The project organization chart, showing lines of responsibility and the specific *CTC* personnel assigned to this project, is presented in Figure 1. A summary of the responsibilities of each *CTC* participant, his/her applicable experience, and his/her anticipated time dedication to the project during testing and reporting is given in Table 7.

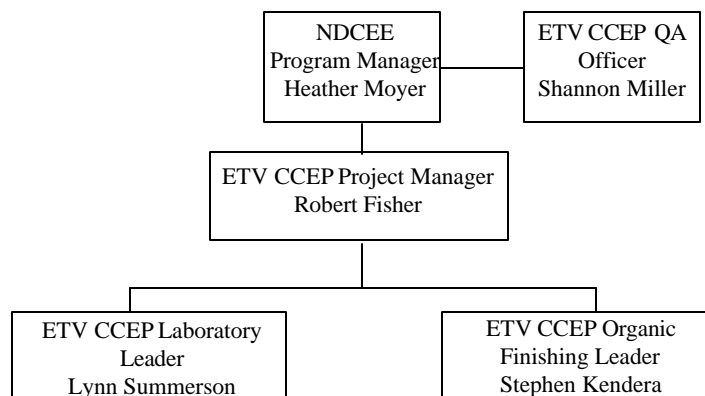


Figure 1. Project Organization Chart

Table 7. Summary of ETV CCEP Experience and Responsibilities

Key CTC Personnel and Roles	Responsibilities	Applicable Experience	Education	Time Dedication
Heather Moyer – NDCEE Program Manager	Manages NDCEE Program Accountable to CTC Technical Services Manager and CTC Corporate Management	Project Manager (10 years)	B.S., Chemical Engineering	1%
Shannon Miller – ETV CCEP QA Officer	Responsible for overall project QA Accountable to NDCEE Program Manager	Quality Mgmt. /ISO 9000 (6 years) Environmental Compliance and ISO 14000 Management Systems (6 years) ISO Internal Auditor (5 years)	B.A., Communications	5%
Rob Fisher – Staff Process Engineer/ ETV CCEP Project Leader	Technical project support Process design and development Accountable to NDCEE Program Manager	Organic Finishing Regulations (10 years) Organic Finishing Operations (10 years) Registered Professional Engineer	M.S., Manufacturing Systems Engineering B.S., Chemical Engineering	60%
Lynn Summerson – ETV CCEP Laboratory Leader/ Statistical Support Staff	Laboratory analysis Accountable to ETV CCEP Project Manager	Industrial and Environmental Laboratory Testing (22 years)	M.S., Chemistry B.S., Chemistry	15%
Stephen Kendera – ETV CCEP Organic Finishing Leader	QC Analysis Accountable to ETV CCEP Project Manager	Organic Finishing Operations (25 years)	N/A	5%

The ETV CCEP personnel specified in Table 7 are responsible for maintaining communication with other responsible parties working on the project. The frequency and mechanisms for communication are shown in Tables 8 and 9.

Table 8. Frequency and Mechanisms of Communications

Initiator	Recipient	Mechanism	Frequency
NDCEE Program Manager and/or ETV CCEP Project Manager	EPA ETV CCEP Project Manager	Written Report Verbal Status Report	Monthly Weekly
ETV CCEP Project Manager	NDCEE Program Manager	Written or Verbal Status Report	Monthly
ETV CCEP Laboratory Leader	ETV CCEP Project Manager	Data Reports	As Generated
ETV CCEP QA Officer	NDCEE Program Manager	Quality Review Report	As Required
EPA ETV CCEP Project Manager	CTC	On-Site Visit	As Needed
Special Occurrence	Initiator	Recipient	Mechanism/ Frequency
Schedule or Financial Variances	NDCEE Program Manager or ETV CCEP Project Manager	EPA ETV CCEP Project Manager	Telephone Call, Written Follow-up Report as Necessary
Major (will prevent accomplishment of verification cycle testing) Quality Objective Deviation	NDCEE Program Manager or ETV CCEP Project Manager	EPA ETV CCEP Project Manager	Telephone Call with Written Follow-up Report

Table 9. Responsibilities During Testing

Position	Responsibility
ETV CCEP Project Manager	Overall coordination of project, testing, reporting, and data reviews
ETV CCEP QA Officer	Audits of verification testing operations and laboratory analyses
Statistical Support	Coordinates interpretation of test results

4.0 QUALITY ASSURANCE OBJECTIVES

4.1 General Objectives

The overall objectives of this ETV CCEP T/QAP are to verify the pollution prevention benefit of the TAGNITE anodizing process and the quality and performance of the resultant finish. These objectives will be met by controlling and monitoring the critical and non-critical factors, which are the specific QA objectives for this T/QAP. Tables 3 and 4 list the critical and non-critical factors, respectively.

The analytical methods that will be used for this evaluation are adapted from ASTM Standards, or industrial standard equivalent. The QA objectives of the program and the capabilities of these test methods for product and process inspection and evaluation are synonymous because the methods were designed specifically for evaluation of the anodizing process properties under investigation. The methods will be used as published, or as supplied, without deviations. The specific methods to be used for this project are attached to this document in Appendix E (ASTM Methods).

4.2 Quantitative Quality Assurance Objectives

Quality assurance parameters such as precision, accuracy, and completeness, are presented in Tables 10 and 11. Table 10 presents the manufacturers' stated capabilities of the equipment used to measure non-critical control factors. The precision and accuracy parameters listed are relative to the true value to which the equipment measures. Table 11 presents the precision and accuracy parameters for the measurement equipment for the critical response factors. Precision and accuracy are determined using duplicate analysis and known standards and/or spikes and must fall within the values found in the specific methods expressed.

The statistical support staff, ETV CCEP QA Officer, and laboratory personnel will coordinate efforts to calculate and interpret the test results.

Table 10. QA Objectives for Precision, Accuracy and Completeness for All Non-Critical Control Factor Performance Analyses

Measurement	Method	Units	Precision	Accuracy	Completeness
Products Involved in Testing (Standard Test Panels and Galvanic Corrosion Coupons)	Visual	N/A	N/A	N/A	100%
Surface Area of Products	Ruler	cm ² (ft ²)	±0.025 (±0.0036)	±0.025 (±0.0036)	100%
Surface Profile of Products	ASME B46.1	Ra	±20%	±3%	100%

Ra is defined as the arithmetic average deviation from the center line of the surface.

Table 11. QA Objectives for Precision, Accuracy and Completeness for All Critical Response Factor Performance Analyses

Measurement^a	Method	Units	Precision	Accuracy^b	Completeness
Chromate or Heavy Metal Content and pH	EPA Methods 200.7 and 218.6	µg/L	±20%	±10%	90%
DFT – Eddy Current	ASTM B 244 (Eddy Current)	mils ^c	20% RPD	10% True Thickness	90%
Taber Abrasion	ASTM D 4060	g?	0.025 ^d	Not reported in ASTM D 4060	90%
Salt Spray, Un-Coated, Scribed (Failure or Score=7)	ASTM B117	Pass/Fail	All Pass or All Fail	N/A	90%
Salt Spray Pit Depth, Un-Coated, Scribed (168 hours)	ASTM B117	Pass/Fail	All Pass or All Fail	N/A	90%
Salt Spray, Sealer, Scribed (1000 hrs or Score=7)	ASTM B117	Pass/Fail	All Pass or All Fail	N/A	90%
Salt Spray, Sealer, Primer/Topcoat, Un-Scribed (1000 hrs or Score=7)	ASTM B117	Pass/Fail	All Pass or All Fail	N/A	90%
Direct Impact, Un-Coated	ASTM D 2794 (Direct & Reverse)	Pass/Fail	All Pass or All Fail	Ranges listed in ASTM D 2794	90%
Direct Impact, Sealer	ASTM D 2794 (Direct & Reverse)	Pass/Fail	All Pass or All Fail	Ranges listed in ASTM D 2794	90%
Galvanic Corrosion Salt Spray, Sealer, Un-Scribed (1000 hrs or Score=7)	ASTM B117	Pass/Fail	All Pass or All Fail	N/A	90%

^a Score = 7 refers to the amount of corrosion considered to be a failure, as determined using ASTM B 117.

^b Accuracy is presented as percent recovery of a standard, unless otherwise noted.

^c 1 mil = 0.001 inch

^d Precision is expressed as the maximum allowable difference for low abrasion resistant coatings at 1000 cycles.

N/A = Not Applicable

RPD - relative percent difference

4.2.1 Accuracy

Standard reference materials, traceable to national sources such as the National Institute for Standards and Technology (NIST) for instrument calibration and periodic calibration verification, will be procured and utilized where such materials are available and applicable to this project. For reference calibration materials with certified values, acceptable accuracy for calibration verification

will be within the specific guidelines provided in the method if verification limits are given. Otherwise, 80-120 percent of the true reference values will be used (see Tables 10 and 11). Reference materials will be evaluated using the same methods as for the actual test specimens. Calculations for precision, accuracy, etc. are contained in Section 10.0.

4.2.2 Precision

The experimental approach of this T/QAP specifies the exact number of Standard Test Panels to be coated. The analysis of replicate Standard Test Panels and Galvanic Corrosion Coupons for all tests at each of the experimental conditions will occur by design. The degree of precision will be assessed based on the agreement of all replicates within a property analysis group.

4.2.3 Completeness

The laboratory strives for at least 90 percent completeness. Completeness is defined as the number of valid determinations expressed as a percentage of the total number of analyses conducted, by analysis type. Samples, which are invalidated due to uncorrectable errors and cannot be re-analyzed, will be considered incomplete.

4.2.4 Impact and Statistical Significance Quality Objectives

All laboratory analyses will meet the accuracy, precision, and completeness requirements specified in Tables 10 and 11. The precision will also be checked on Standard Test Panel or Galvanic Corrosion Coupon replicates to determine whether a nonconformance exists. If any non-conformance from T/QAP QA objectives occurs, the cause of the deviation will be determined by checking calculations, verifying the testing and measuring equipment, and performing reanalysis. If an error in analysis is discovered, reanalysis of a new batch for a given trial will be considered, and the impact to overall project objectives will be determined. If the deviation persists despite all corrective action steps, the data will be flagged as not meeting the specific quality criteria and a written discussion will be generated.

If all analytical conditions are within control limits and instrument and/or measurement system accuracy checks are valid, the nature of any nonconformance may be beyond the control of the laboratory. If, given that laboratory quality control data are within specification and any nonconforming results occur, the results will be interpreted as the inability of the particular technology to produce parts meeting the performance criteria at the given set of experimental conditions.

4.3 Qualitative QA Objectives: Comparability and Representativeness

4.3.1 Comparability

The TAGNITE coating process will be used per TAG's recommendations or conditions otherwise established in agreement with TAG. The data will be comparable from the standpoint that other testing programs could reproduce similar results using a specific T/QAP. The Magnesium anodizing process and environmental performance will be evaluated using EPA, ASTM and other nationally or industry-wide accepted testing procedures. Process performance parameters and cost data will be generated and evaluated according to standard best engineering practices.

Standard Test Panels and Galvanic Corrosion Coupons used in these tests will be compared to the performance data obtained from the baseline tests and to other applicable end-user and industry specifications, such as anticipated corrosion resistance. The specifications will be used to verify the performance of the TAGNITE coating. Additional assurance of comparability comes from the routine use of precision and accuracy indicators as described above, the use of standardized and accepted methods, and the traceability of reference materials.

4.3.2 Representativeness

The limiting factor to representativeness is the availability of a large sample population. Experimental designs will be constructed such that projects will have either sufficiently large sample populations per trial or otherwise statistically significant fractional populations. The tests will be conducted at the paint and equipment supplier-recommended operating conditions. If the test data meets the quantitative QA criteria (precision, accuracy, and completeness), the measurements of the tested samples will be considered representative of the technologies under evaluation and will be used to interpret the outcomes relative to the specific project objectives.

4.4 Other QA Objectives

No other QA objectives have been identified as part of this evaluation.

4.5 Impact of Quality

Due to the highly controllable nature of the Standard Test Panel and Galvanic Corrosion Coupon evaluation methods and predictability of factors affecting the quality of the laboratory testing of panels or coupons, the quality control of Standard Test Panel and Galvanic Corrosion Coupon qualifications is expected to fall within acceptable levels.

Comparison of response factors will be checked for run-to-run process variations. Deviation from quantitative and qualitative QA objectives is not expected.

5.0 SITE SELECTION AND SAMPLING PROCEDURES

5.1 Site Selection

TAG's facility in Grand Forks, ND, will be utilized to apply the TAGNITE treatment to the appropriate samples. Samples will be evaluated before and after treatment. ETV CCEP staff will oversee all phases of the treatment process.

Application of the remaining three treatment processes, the sealer, and the primer/topcoat system are anticipated to occur at CTC's ETF in Johnstown, PA. Evaluation of the performance characteristics for each treatment will be performed in the ETF Environmental Coatings Laboratory. In the event that the necessary equipment is either not available at CTC, other facilities may be utilized to complete the sample preparation or testing.

5.2 Sampling Procedures and Handling

Standard Test Panels and Galvanic Corrosion Coupons will be used in this project. They will be pre-labeled by stamping their ID (identification) number on one side. The experimental design will prepare 192 samples during the verification test. ETV CCEP staff will process the samples according to a pre-planned sequence of stages, which includes those identified in Table 12.

Table 12. Process Responsibilities

Procedure	TAG	ETV CCEP Staff
Provide the Requisite Number and Type of Samples	X	
Inspection of All Samples		X
Numbering of Samples		X
Apply TAGNITE to Samples	X	
Apply Remaining Treatments to Samples		X
Apply Appropriate Coatings to Samples		X
Evaluate the Treated/Coated Samples		X
Conduct Laboratory Testing		X

A laboratory analyst will process the Standard Test Panels and the Galvanic Corrosion Coupons through the ETF Environmental Coatings Laboratory login prior to performing the required analyses.

5.3 Sample Custody, Storage and Identification

The Standard Test Panels and Galvanic Corrosion Coupons will be given unique laboratory ID numbers and logged into the laboratory record sheets. The analyst delivering the samples will complete a custody log indicating the sampling point IDs, sample material IDs, quantity of samples, time, date, and analyst's initials. The samples

will remain in the custody of *CTC*, unless a change of custody form has been completed. The change of custody form should include a signature from *CTC*, the sample ID number, the date of custody transfer, and the signature of the individual to whom custody was transferred.

Laboratory analyses may only begin after each sample is logged into the laboratory record sheets. The laboratory's sample custodian will verify this information. Both personnel will sign the custody log to indicate transfer of the samples from the coating processing area to the laboratory analysis area. The laboratory sample custodian will log the samples into a bound record book; store the samples under appropriate conditions (ambient room temperature and humidity); and create a work order for the various laboratory departments to initiate testing. The product evaluation tests also will be noted on the laboratory record sheet. Testing will begin within several days of receiving the samples.

6.0 ANALYTICAL PROCEDURES AND CALIBRATION

6.1 Facility and Laboratory Testing and Calibration

CTC shall maintain a record of calibrations and certifications for all applicable equipment. A calibration check will be made of the testing and measuring equipment prior to and after the verification test.

6.1.1 Facility Testing and Calibration

Calibration procedures within the ETF organic finishing line and environmental coatings laboratory will follow the applicable standards or manufacturer's recommendations. Certified solutions and reference materials traceable to NIST shall be obtained as appropriate to ensure the proper equipment calibration. Where a suitable source of material does not exist, a secondary standard is prepared and a true value obtained by measurement against a technical-grade NIST-traceable standard.

6.1.2 Laboratory Testing and Calibration Procedures

The analytical methods performed at *CTC* are adapted from standard ASTM, MIL-SPEC, EPA, Association of Official Analytical Chemists (AOAC) and/or industry protocols for similar manufacturing operations. Initial calibration and periodic calibration verification are performed to insure that an instrument is operating sufficiently to meet sensitivity and selectivity requirements. At a minimum, all equipment are calibrated before use and are verified during use and/or immediately after each sample batch. Standard solutions are purchased from reputable chemical supply houses in neat and diluted forms. Where certified and traceable to NIST reference materials and solutions are available, the laboratory purchases these for calibration and standardization. Data from all equipment calibrations and chemical standard certificates from vendors are stored in laboratory files and are readily retrievable. No samples are reported in which the full calibration curve, or the periodic calibration check standards, is outside method performance standards. As needed, equipment will be sent off-site for calibration or certification.

The listing of ASTM Methods used in this verification can be found in Appendix E. All equipment, used for these analyses, is calibrated according to Tables 13 and 14.

6.2 Product Quality Procedures

Each apparatus that will be used to assess the quality of a treatment on a panel or coupon is set up and maintained according to the published reference method's instructions. Actual sample analysis will take place only after setup is verified per the reference method and the equipment manufacturer's instructions. As available, samples of known materials with established product qualities are used to verify that a system is functioning properly. For example, traceable thickness standards are used to calibrate the DFT instrument. Applicable ASTM methods are listed in Appendix E.

6.3 Standard Operating Procedures and Calibration

Tables 13 and 14 summarize the methods and calibration criteria that will be used for the evaluation of the coatings. Each analysis shall be performed per the applicable published methods.

Table 13. Non-Critical Control Factor Testing and Calibration Criteria

Non-Critical Factor	Method	Method Type	Calibration Procedure	Calibration Frequency	Calibration Accept. Criteria
Products Involved in Testing (Standard Test Panels and Galvanic Corrosion Coupons)	Visual	N/A	N/A	N/A	N/A
Surface Area of Standard Test Panels and Galvanic Corrosion Coupons	Ruler	Ruler	Inspect for damage, replace if necessary	With each use	Lack of damage
Surface Profile of Standard Test Panels and Galvanic Corrosion Coupons	ASME B46.1	Stylus	Inspect for damage, replace if necessary	With each use	Lack of damage

N/A = Not Applicable

Table 14. Critical Response Factor Testing and Calibration Criteria

Critical Measurement	Method Number ^a	Method Type	Calibration Procedure	Calibration Frequency	Calibration Accept. Criteria ^b
Chromate and Heavy Metal Content and pH	EPA Methods 200.7 and 218.6	ICP-AES and IC	Comparison to standard blanks or check solutions	Verify calibration after every ten samples	90-110%
DFT	ASTM B 244	Eddy Current	Comparison to NIST-traceable standard per ASTM method	Verify calibration after every three samples	90-110%
Taber Abrasion	ASTM D 4060	N/A	N/A	N/A	N/A
Salt Spray, Un-Coated, Scribed	ASTM B117	Visual	N/A	N/A	N/A
Salt Spray Pit Depth, Un-Coated, Scribed	ASTM B117	Visual	N/A	N/A	N/A
Salt Spray, Sealer, Scribed	ASTM B117	Visual	N/A	N/A	N/A
Salt Spray, Sealer, Primer/Topcoat, Un-Scribed	ASTM B117	Visual	N/A	N/A	N/A
Direct Impact, Un-Coated	ASTM D 2794 (Direct & Reverse)	Visual	N/A	N/A	N/A
Direct Impact, Sealer	ASTM D 2794 (Direct & Reverse)	Visual	N/A	N/A	N/A
Galvanic Corrosion Salt Spray, Sealer, Un-Scribed	ASTM B117	Visual	N/A	N/A	N/A

^a Listing of test methods to be used is provided in Appendix E.

^a (2) As a percent recovery of a standard.

ICP-AES stands for Inductively Coupled Plasma-Atomic Emission Spectrometry

IC = Ion Chromatography

N/A = Not Applicable

6.4 Non-Standard Methods

CTC does not anticipate using any non-standard methods for this verification.

7.0 DATA REDUCTION, VALIDATION, AND REPORTING

7.1 Raw Data Handling

Raw data will be gathered by ETV CCEP staff and recorded onto bench or laboratory data sheets. This data will undergo validation by the ETV CCEP Laboratory Leader and the ETV CCEP Project Manager.

7.2 Preliminary Data Package Validation

The generating analyst will assemble a preliminary data package for each analysis. This package will contain the QC and raw data results, calculations, electronic printouts, conclusions and laboratory sample tracking information. The ETV CCEP Laboratory Leader will review the entire package and may also check sample and storage logs, standard logs, calibration logs, and other files, as necessary, to insure that tracking, sample treatments and calculations are correct. The ETV CCEP Laboratory Leader will hand-check at least 10% of the data calculations. The data reports will be reviewed for clarity and accuracy of transcription. After the package has been peer reviewed in this manner, a preliminary data report will be prepared. The entire package and final laboratory report will be submitted to the ETV CCEP Project Manager.

7.3 Final Data Validation

The ETV CCEP Project Manager shall be ultimately responsible for all final data released from this project. The ETV CCEP Project Manager will review the final results for adequacy to project QA objectives. If the manager suspects an anomaly or non-concurrence with expected or historical performance values, with project QA objectives, or with method specific QA requirements of the laboratory procedures, he will initiate a second review of the raw data and query the generating analyst at CTC and the ETV CCEP Laboratory Leader about the non-conformance. Also, he will request specific corrective action. If suspicion about data validity still exists after internal review of laboratory records, the ETV CCEP Project Manager may authorize a re-analysis. If sufficient sample is not available for re-testing, a re-sampling will occur. If the sampling window has passed, or re-sampling is not possible, the ETV CCEP Project Manager will flag the data as suspect and notify the EPA ETV CCEP Project Manager.

7.4 Data Reporting and Archival

7.4.1 Calculation of DFT Variation

The DFT gauge has a stated accuracy of 0.1 mils. Since the calculated DFT variation is intended for use as quality assurance measures only in this phase of

testing, it is not listed as a Critical Response Factor. Once DFT measurements have been made at several locations on each product (see Appendices A and B), DFT variation will be determined as a function of actual deviations from the average thickness and the standard deviation.

7.4.2 Evaluation of the Technology Applications Group, Inc. TAGNITE Magnesium Anodizing System

The CTC Environmental Laboratory will retain the data packages at least 10 years.

The ETV CCEP Project Manager or the NDCEE Program Manager will forward the results and conclusions to EPA in their regular reports for final EPA approval of the test data. This information will be used to prepare the Verification Report, which will be published by the ETV CCEP. The ETV CCEP project team, TAG, EPA ETV CCEP Project Manager, EPA ETV CCEP QA Manager, EPA technical peer reviewers, and the EPA technical editor will review the Verification Report. The EPA and the NDCEE will then approve the revised document prior to it being published.

7.5 Verification Statement

CTC will also prepare a Verification Statement from the information contained in the Verification Report. After receiving the results and conclusions from the ETV CCEP Project Manager or the NDCEE Program Manager, the EPA will approve the Verification Report and Verification Statement. Only after agreement by TAG, will the Verification Statement be disseminated.

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 Guide used for Internal Quality Program

CTC has established an ISO 9001 operating program for its laboratories and the Demonstration Factory within the ETF. The laboratory is currently establishing a formal quality control program for its specific operations. The format for laboratory QA/QC is being adapted from several sources, as listed in Table 15.

Table 15. *CTC* Laboratory QA/QC Format Sources

Document	Reference Source
General Requirements for the Competence of Calibration and Testing Laboratories	ISO Guide 25, ISO Quality Programs
Critical Elements for Laboratories	Pennsylvania Department of Environmental Protection
Chapter One, Quality Control	SW-846, EPA Test Methods
Requirements of 100-300 Series of Methods	EPA Test Methods
Handbook of Quality Assurance for the Analytical Chemistry Laboratory, 2 nd Edition	James P. Dux

8.2 Types of QA Checks

The ETF laboratory at *CTC* shall follow published methodologies, wherever possible, for testing protocols. Laboratory methods shall be adapted from Federal specifications, military specifications, ASTM test methods, and supplier instructions. The ETF laboratory adheres to the QA/QC requirements specified in these documents. Each *CTC* facility that uses standard test products implements its own level of QA/QC. *CTC*'s laboratory within the ETF will perform the testing and QA/QC verification as outlined in Tables 10 and 11 (Precision, Accuracy, Completeness) and Tables 13 and 14 (Calibration); therefore, these tables should be referred to for the method-specific QA/QC that will be performed.

8.3 Basic QA Checks

During each test, laboratory staff will complete an internal process QA checklist to ensure that the appropriate parts, standard test products, samples, and operating conditions are used. The laboratory also monitors its reagent-deionized water to ensure it meets purity levels consistent with analytical methodologies. The filters are replaced quarterly before failures are encountered. When failures do occur, samples are not processed until the filters are replaced. Blank levels must not exceed minimum detection levels for a given parameter to be considered valid for use.

Thermometers are checked against NIST-certified thermometers at two temperatures. The laboratory checks and records the temperatures of sample storage areas, ovens, hot plate operations, and certain liquid baths using thermometers.

Balances are calibrated by an outside organization using standards traceable to NIST. *CTC* also performs in-house, periodic verifications with ASTM Class 1 weights. The ETF laboratory maintains records of the verification activities and calibration certificates. The laboratory analyst also checks the balances prior to use with ASTM Class 1 weights.

Reagents purchased directly by the laboratory are American Chemical Society grade or better. Reagents, dated on receipt and when first opened, are not used beyond their certified expiration dates.

Laboratory waste is segregated according to chemical classifications in labeled containers to avoid cross-contamination of samples.

Worksheets will be used to record the various data required by this project. Each worksheet will contain the name of the process with which it is associated, as well as blanks for the date, the name of the staff member immediately responsible for carrying out the measurements, and the associated data.

At the end of each experiment, or at the end of the workday, all worksheets for that date will be collected, double-checked for completeness, and filed into a dated folder. These data will be entered into a computer for analysis.

8.4 Specific Checks

CTC laboratory personnel will also check any referenced materials and equipment as available and specified by the referenced methodology and/or the project-specific QA/QC objectives. *CTC* laboratory records will be maintained with the sample data packages and/or in centralized files, as appropriate. To insure comparability, the *CTC* laboratory will carefully control process conditions and perform product evaluation tests consistently for each specimen. The specific QA checks listed in Tables 10, 11, 13, and 14 provide the necessary data to determine if process control and product testing objectives are being met. ASTM, Federal, and Military methods that are accepted in industry for product evaluations, and supplier-endorsed methods for process control, are used for all critical measurements, thus satisfying the QA objective. A listing of the ASTM methods that will be used for this T/QAP is included in Appendix E.

The ETV CCEP QA Officer, who is independent of the ETV CCEP project management, will perform QA audits of the testing and laboratory analyses to supplement *CTC*'s QC checks. These audits will check that processes are completed as per the approved written documentation, both internal and external. The QA audits will also check that the laboratory data is handled properly.

The calibration checks generally consist of calibrating the equipment (if applicable), checking the calibration against a secondary standard, analyzing samples, rechecking the calibration, analyzing more samples, etc. The calibration is also checked against the secondary standard at the completion of an analysis series. If, at any time, the equipment falls out of calibration, all samples analyzed since the last good calibration check will be re-analyzed after the equipment is re-calibrated.

9.0 PERFORMANCE AND SYSTEM AUDITS

CTC has developed a system of internal and external audits to monitor both program and project performance. These include monthly managers' meetings and reports, financial statements, EPA reviews and stakeholders' meetings, and In-Progress Reviews. The ETF laboratory also analyzes performance evaluation samples to maintain Pennsylvania Department of Environmental Protection Certification.

ISO Internal Audits

CTC has established its quality system based on ISO 9000 and 14000 and has implemented a system of ISO internal audits. This information will be used for internal purposes.

On-Site Visits

The EPA Project Manager may visit *CTC* for an on-site visit during the execution of this project. All project, process, quality assurance, and laboratory testing information will be available for review.

EPA Audits

The EPA may periodically audit *CTC* during this project. All project, process, quality assurance, and laboratory testing information will be made available per the EPA's auditing procedures.

Technical Systems Audits

A listing of all equipment used, laboratory measuring and testing devices and procedures, a copy of the currently approved ETV QMP, and the currently approved ETV CCEP QMP will be given to the ETV CCEP QA Officer for this project. The QA Officer will conduct an initial audit, and additional audits thereafter according to the ETV CCEP QMP, of demonstration and testing activities. The results of this activity will be forwarded to EPA in reports from the NDCEE Program Manager or the ETV CCEP Project Manager.

Audits of Data Quality

Peer review in the laboratory constitutes a process whereby two analysts review raw data generated at the bench level. After data are reduced, they undergo review by ETV CCEP laboratory management. For this T/QAP, the ETV CCEP QA Officer will spot-check 10% of the project data by performing a total review from raw to final results. This activity is performed in addition to the routine ETV CCEP review of all data. Records will be kept to show which data have been reviewed in this manner.

10.0 CALCULATION OF DATA QUALITY INDICATORS

10.1 Precision

Duplicates will be performed on separate, as well as on the same sample source, depending on the method being employed. In addition, the final result for a given test may be the arithmetic mean of several determinations on the product or matrix. In this case, duplicate precision calculations will be performed on the means. The following calculations will be used to assess the precision between duplicate measurements.

$$\text{Relative Percent Difference (RPD)} = [(C1 - C2) \times 100\%] / [(C1 + C2) / 2]$$

where: C1 = larger of the two observations
C2 = smaller of the two observations

$$\text{Relative Standard Deviation (RSD)} = (s/y) \times 100\%$$

where: s = standard deviation
y = mean of replicates.

10.2 Accuracy

Accuracy will be determined as percent recovery of a check standard, check sample or matrix spike.

For matrix spikes and synthetic check samples:

$$\text{Percent Recovery (\%R)} = 100\% \times [(S - U)/T]$$

where: S = observed concentration in spiked sample
U = observed concentration in un-spiked sample
T = true value of spike added to sample.

For standard reference materials (srn) used as calibration checks:

$$\% R = 100\% \times (C_m / C_{srn})$$

where: C_m = observed concentration of reference material
C_{srn} = theoretical value of srn.

10.3 Completeness

$$\text{Percent Completeness (\%C)} = 100\% \times (V/T)$$

where: V = number of determinations judged valid
 T = total number of determinations for a given method type.

10.4 Project Specific Indicators

Process control limit: range specified by supplier for a given process parameter.

11.0 CORRECTIVE ACTION

11.1 Routine Corrective Action

Routine corrective action will be undertaken in the event that a parameter in Tables 10, 11, 13, or 14, is outside the prescribed limits specified in these tables, or when a process parameter is beyond specified control limits. Examples of non-conformances include invalid calibration data, inadvertent failure to perform method-specific QA tests, process control data outside specified control limits, and failed precision and/or accuracy indicators. Such non-conformances will be documented on a standard laboratory form. Corrective action involves taking any necessary steps to restore a measuring system to proper working order and summarizing the corrective action and results of subsequent system verifications on a standard form. Some non-conformances will be detected during analysis or sample processing, and can be rectified in real time at the bench level. Others may be detected following completion of processing trial and/or sample analyses. Typically, these types of nonconformances will be detected at the ETV CCEP laboratory management level of data review. In all cases of nonconformance, the ETV CCEP Project Manager will consider repeating the sample analysis as one method of corrective action. If insufficient sample is available, or the holding time has been exceeded, complete reprocessing may be ordered to generate new samples. Reprocessing will only be performed if the ETV CCEP Project Manager determines that the nonconformance will jeopardize the integrity of the conclusions to be drawn from the data. In all cases, a nonconformance will be rectified before sample processing and analysis continues. If corrective action does not restore the production or analytical system causing a deviation from the ETV CCEP QMP, CTC will contact the EPA ETV CCEP Project Manager. In cases of routine nonconformance, EPA will be notified in the NDCEE Program Manager's or ETV CCEP Project Manager's regular report to the EPA ETV CCEP Project Manager. A complete discussion will accompany each nonconformance.

11.2 Non-Routine Corrective Action

While not anticipated, internal audits by the ETV CCEP QA Officer, or onsite visits by the EPA ETV CCEP Project Manager, may identify nonconformances of the requirements stated in the ETV CCEP QMP. In the event that nonconformances are detected by bodies outside the ETF laboratory organizational unit, as for routine nonconformances, these problems will be rectified and documented prior to processing or analyzing further samples or specimens.

12.0 QUALITY CONTROL REPORTS TO MANAGEMENT

As shown in Table 7 (Summary of ETV CCEP Experience and Responsibilities), the ETV CCEP QA Officer is independent of the ETV CCEP project management team. It is the responsibility of the ETV CCEP QA Officer to monitor ETV CCEP verification tests for adherence to project specific QMPs and T/QAPs. The ETV CCEP QA Officer will audit the operation records, laboratory records, and laboratory data reports and provide a written report of his findings to the ETV CCEP Project Manager and to the ETV CCEP laboratory management. The ETV CCEP Project Manager will insure that these reports are included in his report to EPA. The ETV CCEP laboratory management will be responsible for achieving closure on items addressed in the report. Specific items to be addressed and discussed in the QA report include the following:

- ? General assessment of data quality in terms of general QA objectives in Section 4.1
- ? Specific assessment of data quality in terms of quantitative and qualitative indicators listed in Section 4.2 and 4.3
- ? Listing and summary of all non-conformances and/or deviations from the ETV CCEP T/QAP
- ? Impact of non-conformances on data quality
- ? Listing and summary of corrective actions
- ? Results of internal/external QA audits
- ? Closure of open items from last report or communications with EPA in current reporting period
- ? Deviations or changes in the ETV CCEP QMP
- ? Limitations on conclusions, use of the data
- ? Planned QA activities, open items for next reporting period.

13.0 REFERENCES

The following documents are referenced in this T/QAP:

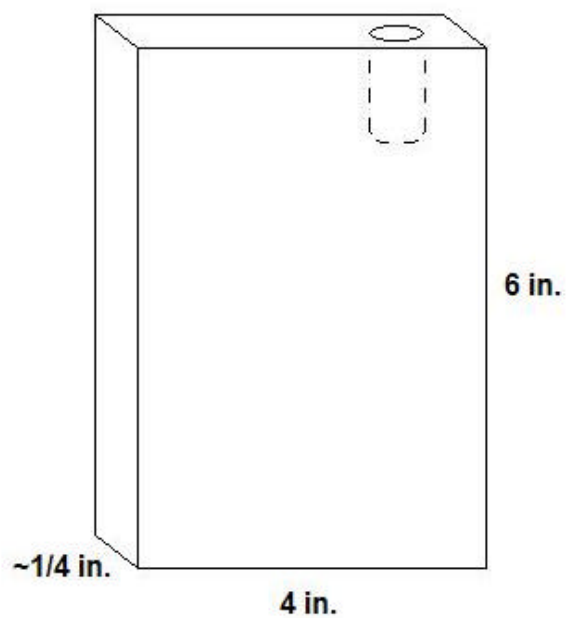
American Society for Quality Control. American National Standard Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs. ANSI/ASQC E4-1994, E4. American Society for Quality, 1994.

ETV Program QMP

Environmental Technology Verification Program Quality and Management Plan, EPA/600/R-03/021, Cincinnati OH: U. S. Environmental Protection Agency, 2002.

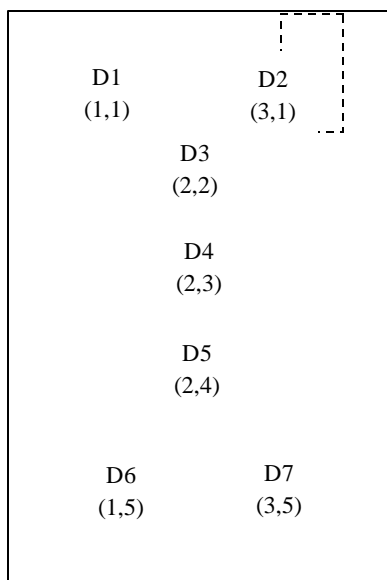
APPENDIX A

Standard Test Panels



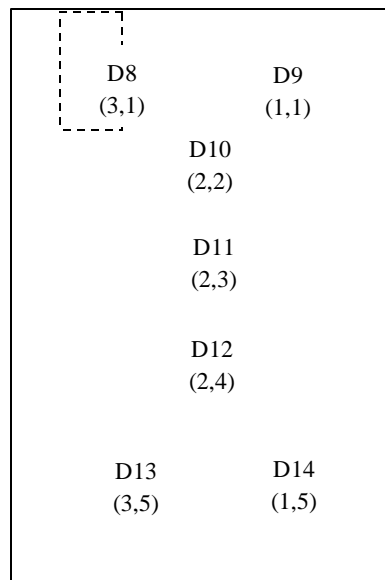
DFT Measurement Sites on the Standard Test Panels

(0,0)



Side A

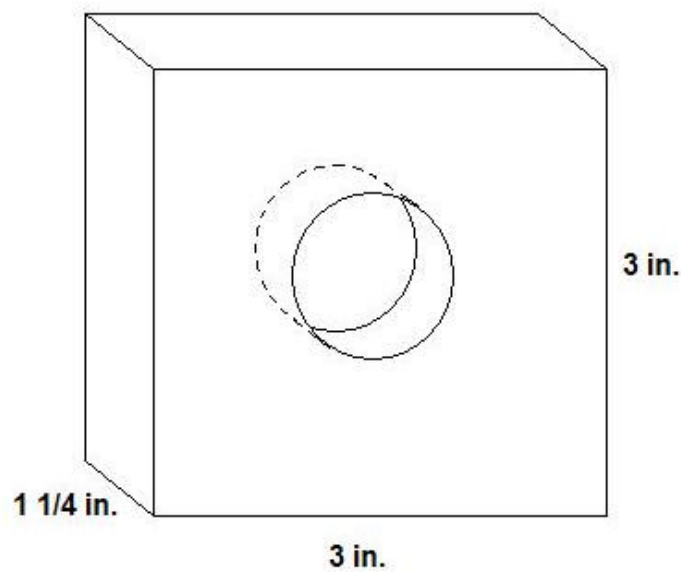
(0,0)



Side B

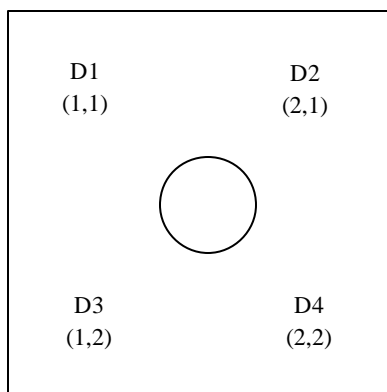
APPENDIX B

Galvanic Corrosion Coupons



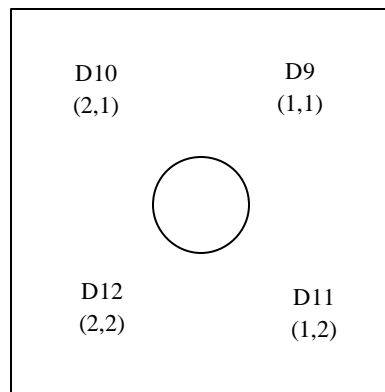
DFT Measurement Sites on the Galvanic Corrosion Coupons

(0,0)



Side A

(0,0)



Side B

Note: Sites D5, D6, D7, and D8 are located in the center of each of the four edges.

APPENDIX C

TAGNITE Product Specifications



APP_C.pdf

APPENDIX D

Baseline Treatments Product Specifications



APP_D.pdf

APPENDIX E

ASTM Methods

ASTM Methods

- ASME B46.1 -- Surface Texture (Surface Roughness, Waviness, and Lay)
[Referenced/Supplemental Methods: ASME Y14.36M, ASME D89.6.2, and ASME Y14.5M]
- ASTM B 117 -- Standard Test Method of Salt Spray (Fog) Testing [Referenced/Supplemental Methods: ASTM B 368, ASTM D 1193, ASTM D 1654, ASTM D 609, ASTM E 691, ASTM E 70, and ASTM G 85]
- ASTM B 244 -- Measurement of Thickness of Anodic Coatings on Aluminum and of Other Nonconductive Coatings on Nonmagnetic Basis Metals with Eddy-Current Instruments [Referenced/Supplemental Methods: ASTM B 499]
- ASTM D 2794 -- Standard Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact) [Referenced/Supplemental Methods: ASTM D 609, ASTM D 823, and ASTM D 1186]
- ASTM D 4060 -- Standard Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser [Referenced/Supplemental Methods: ASTM D 823, ASTM D 968, ASTM D 1005, ASTM D 1186, ASTM D 1400, ASTM D 2240, and ISO 7784-2}
- EPA Method 200.7 -- Determination of Metals and Trace in Water and Wastes by Inductively Couple Plasma-Atomic Emission Spectrometry
[Referenced/Supplemental Methods: ASTM D 1193]
- EPA Method 218.6 -- Determination of Dissolved Hexavalent Chromium in Drinking Water, Groundwater, and Industrial Wastewater Effluents by Ion Chromatography
[Referenced/Supplemental Methods: EPA Method 1636, EPA Method 1669, EPA Method 200.2, and ASTM D 1193]